LOW TEMPERATURE BATTERY
(NEW MAGNESIUM ANODE STRUCTURE)

REPORT NO. 4

CONTINUATION OF CONTRACT NO. DA 36-039-AMC-00021 (E)
ARMY MATERIEL COMMAND
CONTRACT NO. DA 36-039 AMC-03369 (E)

DEPARTMENT OF THE ARMY
TASK NO. 1C6-22001-A-053-02

SECOND SEMI-ANNUAL REPORT
1 APRIL 1964 TO 30 SEPTEMBER 1964

U. S. ARMY ELECTRONICS LABORATORIES
FORT MONMOUTH, NEW JERSEY

CARIBBEAN TRADING CORPORATION
FORT LAUDERDALE, FLORIDA

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Army Materiel Command Contract No. DA 36-039 AMC-03369(E)

USAEL Technical Guidelines for PR & C No. 62-ELP/D-4217

Department of the Army Task No. 1C6-22001-A-053-02

SECOND SEMIANNUAL REPORT

1 April 1964 to 30 September 1964

Object:

a) Research and development of a new type non-reserve magnesium "D" Size cell utilizing a reversed electrode type of dry battery structure covered by U. S. Patent No. 2,903,499.

b) Manufacture and assembly of magnesium "D" Size cells incorporating above mentioned R and D work for testing and evaluation by Contractor and Subcontractor, The Dow Chemical Company, Midland, Michigan.

Report prepared by:

Rodolfo R. Balaguer

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PURPOSE

Research work directed towards the development of a new dry cell utilizing the Balaguer reverse electrode type dry cell battery structure, leading to the construction of 500 magnesium "D" size cells to be delivered to the U. S. Army Electronics Laboratories, incorporating the best results obtained by the contractor.

ABSTRACT

Investigations of shelf life and low temperature performance consisted principally of two shelf and two experimental design programs. Investigations of anode pitting and slow voltage recovery of the cell comprising seven screening programs, involving various modifications and combinations of cell components, were undertaken. Cleaning of the anode after forming with Dow 21 pickle minimized the pitting. The plastic cell closure has markedly improved the shelf life of the Balaguer "D" size cell. Employing a seamless steel jacket in place of a seam steel jacket reduced the radial expansion during cell discharge.

CONFERENCES

(I) Subject: Pitting of the Anode

Organizations Represented: Power Sources Div., USAEL
(Mr. Donald Wood)

The Dow Chemical Co.
(Mr. J. L. Robinson)

Caribbean Trading Corp.
(Messrs. R. Balaguer and
J. D. Hedges)

Place: Atlantic City, New Jersey

Date: May 21, 1964

Conclusions: Two Screening Programs were outlined

(II) Subject: Shelf Life of the Cell

Organizations Represented: Power Sources Div, USAEL
(Mr. Donald Wood)

The Dow Chemical Company
(Mr. J. L. Robinson)

Caribbean Trading Corp.
(Messrs. R. Balaguer,
J. D. Hedges & G. Perez)

Place: Caribbean Trading Corporation
Fort Lauderdale, Florida

Date: July 14, 1964

Conclusions: Screening Programs were outlined to isolate the causes of the pitting and high time delay.

A - EQUIPMENT

The same equipment used in the first half of this contract for the development work was used in the second half.

Jigs and dies were made to facilitate the construction of a seamless tinned steel jacket.

B. EXPERIMENTAL DESIGN PROGRAM NO. 4

Presented in Tables I and II are data obtained after high temperature storage from cells fabricated for Experimental Design Program No. 4 described in Report No. 3. The purpose of this experiment was to determine the relative influences of mix wetness, mix height, and consolidation pressure.

As was previously disclosed, the cells in this program were made with a mechanically attached plastic bottom which had already proven to be inadequate in providing cells with acceptable high temperature storage characteristics. In further evaluations, cells with the more reliable cast-in-place plastic bottom will be used.

Since radial expansion caused the seams of the tinned steel, outer jackets to pull apart, a seamless outer jacket will be evaluated.

In reference to Table I, it should be noted that the results of discharging cells at 7-1/2 ohms continuous after one month storage at 130°F. were startling because of the large increase in capacity obtained in every case. Quoting Dow: "The average increase for the eight (8) lots was 28.2% with capacities reaching as high as 42 hours. While these cells had plastic closures, the reason for the large capacity increases is not known. In general, capacities again followed the mix weights, and wetness has not significant effect. The radial expansion with these high capacities is of concern, which emphasizes the need for a stronger jacket."

The cell discharge data at low temperature obtained at the U. S. Army Electronics Laboratories for this program was received. Due to the high time delay observed in these cells and the corrosion of the contact at the closure, no attempt was made to evaluate the effect of the parameters studied.

C - EXPERIMENTAL DESIGN PROGRAM NO. 5

As mentioned in our Report No. 3, a batch of seventy-two (72) cells was assembled for the Experimental Design Program No. 5 for low temperature evaluation of various mixed bromides as cell electrolyte. Table XVI of this same report presents the batch description.

A summary of initial low temperature discharge data was received from the USAEL and is shown in Table No. III.

The evaluation made by Dow of the data presented in Table III reads as follows:

"No service above end voltage was obtained in the 2.25 ohms test at minus 20°F. Mixed bromides had drastically lower delays than $MgBr_2$ in otherwise comparable cells, with little difference in capacities. Of the mixed bromides tested, the best all-around performance was obtained with Mg/Sr mixture having a normality ratio of 2/1 (214 g/l $MgBr_2$ plus 144 g/l $SrBr_2$). Further work around this composition is recommended with quantitive measurements of delay."

D - EXPERIMENTAL DESIGN PROGRAM NO. 6

Experimental Design Program No. 6 was conducted to evaluate magnesium perchlorate electrolyte in cells tested at low temperatures. Cell specifications were as follows:

Cathodes: Type M manganese dioxide, 88-1-3-8, wet 620 ml. of electrolyte/1000 gm. dry mix, 55 gms. of mix consolidated at 40 lbs/in^2.

Anode: 0.055" magnesium AZ 21 x 1

Separator: Kraft paper coated on both sides with 5% Methocel solution.

Structure: Fiberglass tape wrapping, steel jacket with seam, and plastic closure.

Batch No.	$Mg(ClO_4)_2$ Normality	Concentration grams/liter	Na_2CrO_4 grams/liter
1	2.0	223	0
2	3.5	391	0
3	5.0	558	0
4	2.0	223	0.25
5	3.5	391	0.25
6	5.0	558	0.25

Table IV shows initial data as reported by Dow. Table V shows the low temperature data as received from the U. S. Army Electronics Laboratories. Due to the high time delay observed, no effort was made to analyze the effect of the variables under study. As will be noted later, the anodes were pitted. This pitting accentuates the time delay and impairs the overall performance of the cells.

The capacity retention data and influence of storage on delayed action indicates that, aside from the leakage evident in cells subjected to high temperature storage when the steel closure was used (which has been solved by using the plastic closures), there are other sources of trouble that impair the shelf life and time delay.

With depolarizer mix from Batch No. 6 of Experimental Design Program No. 6 a shelf life program was run. These cells contained 5N $Mg(ClO_4)_2$ and all other fabrication parameters employed in Batch No. 6. Twenty-four cells were fabricated. Eighteen (18) of these cells were stored at 130°F. Presented in Table No. VI are the results obtained from the first 5 months. The other six were subjected to storage at 165°F. and data obtained thru 3 months storage is shown in Table No. VII.

The time delay on fresh cells thru 7.5 ohms to 1.0 V. varied from 1 to 3 seconds while cells stored 5 months at 130°F. gave an average delay of four minutes to 1.0 volts thru 7.5 ohms.

Additional data on cells having the cast-in-place plastic bottom (welded contact bottom closure) which showed good retention capacity after 15 and 30 days storage at 165°F. as reported in Semiannual Report No. 3 is shown in Table VIII.

Results for an equal batch of cells stored at 130°F. are shown in Table IX. After 3 months, one cell had a time delay of 6 seconds, while the other had one second. The seam of the jackets was opened by the expansion in both tests during discharge. This problem has been cured by the use of the seamless steel jacket.

The high retention capacity after 3 months at 130°F. with no expansion of the cell on shelf confirms that the plastic bottom has curtailed the major portion of capacity loss on storage. These cells were made with the steel jacket with seams.

While the test results shown in Table VIII at 165°F. are satisfactory for such high temperature, it is equally true that the batteries dried out by the end of the third month. Nevertheless, the same contruction tested at 165°F. as shown in Table VII with the perchlorate of Batch #6 from Experimental Program No. 6, stood up unusually well for three months.

E - SHELF PROGRAM NO. 3

Fifty-three (53) cells were assembled in order that the cast-in-place plastic bottom closure could be evaluated with cells containing aqueous $MgBr_2$ electrolyte.

The specifications for these cells were:

Anode: 0.055" magnesium AZ 21 x 1

Cathodes: 55 gms. mix, type M manganese dioxide, 88-1-3-8, wet 550 ml. electrolyte/1000 gm. dry mix consolidated at 40 lbs/in^2.

Electrolyte: 250 g/l $MgBr_2$ plus 0.25 g/l Na_2CrO_4

Separator: Kraft paper coated on both sides with 5% Methocel in water.

Structure: Fiberglass tape wrapping, steel jacket with seam, and plastic closure.

The initial data is shown in Table X.

When cells from Shelf Program No. 3 were dissected, severe open circuit corrosion was evident. The most likely cause of this corrosion appears to be mix shorting. Since this parasitic corrosion could greatly alter the performance, no attempt was made to analyze the effect of the variables under study in the program.

F - SCREENING PROGRAMS NOS. 1 & 2 - EXPERIMENTAL DESIGN PROGRAM NO. 7

Screening Program No. 1 (Separator Study)

The purpose of this program is to determine if thicker separator would limit the parasitic corrosion by curtailing mix shorting.

The general "D" size cell specifications were: Type M manganese dioxide, wet 550 ml. of electrolyte/1000 gm. dry mix; 0.055" magnesium AZ21 x 1; 40 lbs/in^2 consolidation pressure; plastic closure; fiberglass tape wrapping; and new seamless steel jacket which had been tested at 7-1/2 ohm breakdown test without showing cracks or distortion after four weeks.

The four conditions were as follows:

1. Single separator
2. Two separators
3. Three separators
4. Hand wrapped single separator (original method of covering anode with four strips of paper with edges overlapping).

Fifty-two grams of mix were used for conditions 1, 2 and 4 and 50 grams for condition 3. When the cells were about fourteen days old, two cells from each of these cell lots were dissected and the anode inspected after measuring flash currents, initial voltages, and delayed actions. The data are summarized in Table No. XI. The condition of the respective anodes indicates that the severe open circuit corrosion

being encountered is due to cathode mix shorting. This conclusion was based on the observation that the magnitude of the corrosion was drastically reduced in the cell lots employing two or three layers for the separator when the new method of applying was employed or by reverting to the original method of applying a one-layer separator described in Report No. 9. However, even with the multi-layer separator or the original method of application, excessive pitting still indicated a degree of mix shorting, but it could conceivably be associated with the metal itself or the surface treatment. With two of these anodes, mix short pits were definitely observed along the butt joint indicating separator tearing on insertion of the anode.

With all cells from Screening Program No. 1, very slow initial voltage recovery was observed. This type of "delayed action" is most likely due to one or more of the following causes:

(1) High initial anode to cathode resistance because of the lack of a compacting step after anode insertion.

(2) A highly protective film which is difficult to break down, resulting from the currently employed final anode cleaning step.

(3) Drying out of the separator due to a poor seal.

Screening Program No. 2 (Consolidated Study)

In order to determine if the method of fabricating the cell by driving the anode into the mix was contributing to the parasitic corrosion by creating ruptures in the separator, a group of cells were made similar to Screening Program No. 1, except that the cathode mix was

consolidated after, instead of prior to, anode insertion. The external cell contact was accomplished through a thin strip of tinned steel welded to the magnesium anode. The cells were sealed with self-curing resins.

The discharge characteristics obtained (Table No. XII) and the open circuit corrosion patterns were comparable to the corresponding data and corrosion patterns observed for Program I. Based on these observations, the conclusions are that the current standard assembly technique results in adequate mix consolidation and that anode insertion into the consolidated mix does not increase possible cathode mix penetration of the separator to cause internal shorting.

Experimental Design Program No. 7

The program was run on the assumption that increasing the separator thickness would limit corrosion condition. A 2^3 experiment was run with the following specifications to determine the influence of the three variables:

Cup: Regular

Structure: Fiberglass tape wrapping, seamless steel jacket, plastic closure, 2 ply kraft with asphalt insulating washer.

Separator: Three layers kraft paper coated with Methocel.

Consolidation: 40 lbs/in^2 pressure

Variables: Electrolytes

A - 250 g/l $MgBr_2$ - 0.25 g/l Na_2CrO_4

B - 280 g/l $Mg(ClO_4)$ - 9.25 g/l Na_2CrO_4

Mix Wetness
A - 490 ml. of electrolyte/1000 gms dry mix
B - 550 ml. of electrolyte/1000 gms dry mix

Mix Weight
A - 40 grams
B - 50 grams

The initial data for Experimental Design Program No. 7 are shown in Table XIII. No attempt was made to evaluate these data and further testing was suspended. These cells exhibited severe open circuit corrosion and also in several of the cells the bottom closure pushed out allowing electrolyte leakage. Puncturing of selected cells showed a substantial gas pressure build-up indicating too tight a seal. A double cardboard washer is now being employed to increase the hydrogen venting capacity.

G - SCREENING PROGRAMS NOS. 3, 4, 5, 6 and 7

In view of the cell anode corrosion still evident in Screening Programs No. 1 and 2 and Experimental Plan No. 7, additional information was required to determine the factors causing the corrosion. In order to further isolate the source of trouble, five screening programs were run, numbers 3, 4, 5, 6 and 7.

A very probable source of the difficulty was traced to the anode cleaning and forming methods. All cells assembled up to and through Experimental Design Program No. 7 had anodes which were cleaned by immersing, after forming, in a hot chromic acid solution composed of 250 g/l chromic oxide and 0.1 g/l silver nitrate and rinsing in fresh running water, as suggested by Dow Chemical. The magnesium anode material supplied by Dow had been treated in the Dow 21 bath, which leaves a chromate film. Heating the anodes above 450°F. during anode forming operations could cause reduction of the chromate ion, leaving cathodic chromium metal on the surface of the magnesium. The hot chromic acid cleaner described above would not remove such metallic deposits and, further, this bath does activate the surface in respect to corrosion by forming local passive or cathodic areas on the surface.

In the future, the magnesium sheet will be received without the Dow 21 chromium film and the anodes will be cleaned after forming with the Dow 21 pickle, a bath containing chromic acid, ferric nitrate and potassium flouride. This procedure will eliminate the possibility

of forming cathodic chromium on the anode surface.

The cells for Screening Programs Nos. 3, 4, 5, 6 and 7 employed 1/32 carbon cup; 0.055" magnesium; type M manganese dioxide, 88-1-3-8, wet 550 ml. of electrolyte/1000 gms. dry mix, 250 g/l $MgBr_2$ plus 0.25 g/l Na_2CrO_4; fiberglass tape wrapping; and steel seamless jacket.

Screening Program No. 3

The purpose of this program is to attempt to remove the cathodic chromium deposit which may be formed during the anode cleaning process with Dow 21, a magnesium cleaning bath.

A. Separator: One layer kraft paper coated with Methocel.

B. Anode: Old metal supplied with Dow 21 film. After forming, anodes should be cleaned in Dow 21 for one, three and five minutes.

The cells from Screening Program No. 3 were stored at 130°F. for 1, 2, 5 and 9 weeks.

After the first week, no pitting was observed in the anodes pickled for three and five minutes with fine pitting appearing in those pickled for one minute.

After two weeks, the corrosion pattern was almost identical.

After five weeks, the pitting increased markedly in the cells with anodes pickled for one minute; it started showing in the cells with anodes pickled for three minutes, but none was visible in cells with anodes pickled for five minutes.

After nine weeks, general pitting was observed in all the anodes. The time delay (4 ohms) at the end of the nine week period increased to 3 seconds in the cells with anodes pickled for five minutes, to 17 seconds in the cells with anodes pickled for three minutes and to 20 seconds in the cells with anodes pickled for one minute.

Screening Program No. 4

The objective of Screening Program No. 4 was to determine if the mix shorting which may have been causing the anode corrosion during stand could be curtailed by employing various thicknesses of separators and to evaluate the presently employed consolidation technique.

A. Anode: Metal supplied without Dow 21 and treated, after forming, for 3 minutes in the Dow 21.

B. Separator: Kraft paper with 5% Methocel in water.

1. Single separator
2. Two separators
3. Three separators
4. Hand wrapped single separator (original method of covering anode with four strips of paper with edges overlapping).

In filling the cell, approximately 10 gms. of mix were tamped in the bottom, the anode inserted, and then the cell filled with the bulk of the mix using 10 gm. portions and tamping between additions. The total amount of mix was 50 gms. At present no data is available on this program.

Screening Program No. 5

The objective of this program was to determine if magnesium sheet not treated with Dow 21 prior to forming would provide an acceptable anode and if severe anode cleaning would be required in order to obtain a stable cell.

A. Separator: One layer kraft paper with Methocel.

B. Anode: AZ 21 magnesium alloy supplied without Dow 21. After forming, the following anode treatments were applied.

1. 3 minutes in Dow 21
2. 20 seconds in 70% H_2SO_4 at 120°F.
3. 20 seconds in 70% H_2SO_4 at 120°F. plus 3 minutes in the Dow 21.

The anodes of cells stored at ambient temperatures for two weeks with a final Dow 21 chromate film showed only superficial open circuit corrosion and voltage recovery times of the order of 0.4 seconds. Anodes with the sulphate film showed open circuit etching but delayed action of the order of 20 seconds. The high delay with the sulphate film was not unexpected.

Other cells were stored at 130°F. for 1, 3, 6 and 10 weeks. In the first week, none of the anodes showed any pitting.

The anodes made with metal supplied without the Dow 21 and treated for 20 seconds in 70% H_2SO_4 only, had no pitting after one week and presented a better surface than those using metal supplied with the Dow 21 and treated, after forming, for one minute in Dow 21 as in Screening Program No. 3.

In the third week, there was no visible pitting in any case.

After six weeks, general fine pitting appeared in all anodes, the pitting being more pronounced in the anodes treated in both H_2SO_4 and Dow 21.

After 10 weeks, all anodes showed general fine pitting with the pitting being more marked in the anodes treated with Dow 21 only. This is in contradiction to the results for six weeks.

The time delay at the end of the ten weeks was: 12 seconds for cells with anodes treated with Dow 21 only, and with both H_2SO_4 and Dow 21. The delay for anodes treated in H_2SO_4 only was four seconds average.

Screening Program No. 6

In this program two methods of forming the cell anodes were evaluated. Presently the anodes are formed by applying force to a heated steel die with a hammer. This method of fabrication results in scratches in the anode surface, a possible cause of parasitic corrosion. In order to avoid these scratches a rolling mill employing Haynes Alloy 25 rollers has been designed. The rolled anodes do not show any scratches on their surface.

A. Separator: One layer kraft paper with 5% Methocel.

B. Anode: New metal supplied without the Dow 21. After forming, the anode was cleaned for 3 minutes in the Dow 21.

The following cell data were obtained:

	Initial Cell 1047	One week at 113°F. Cell No. 1048
Open circuit voltage	1.90	1.80
Closed circuit voltage 7-1/2 ohms	1.80	1.70
Seconds to 1 volt	0.3	1.6
Flash current amperes	8.4	6.6

The anodes of dissected cells showed that the pitting was of the same order in both anode forming techniques for cells stored one month at 113°F. The scratches made by the steel die appear to have no effect on the parasitic corrosion.

Screening Program No. 7

The objective of this program is to determine the influence of various separator types on the corrosion.

Anode: Metal supplied without the Dow 21 pickle, but treated for three minutes in Dow 21 after forming.

Separator: 5% Methocel coated

1. Fiberglass cloth
2. Batiste fabric
3. Gauze
4. Methocel paper, commercial type, no inhibitor

Cell data obtained are shown in Table XIV. The visual observations of the corrosion pattern conformed to the cell data. Shallow unacceptable crater type pitting was observed with the Methocel paper indicating some undesirable, perhaps stray mercury, contaminate in the Methocel. No signs of mix shorting was observed with the Methocel paper separator. The reason for the lack of voltage with

cell No. 897 could not be found. The anodes with the fiberglass separator perforated as a result of mix shorting which was clearly visible. The anode corrosion pattern with the sterile gauze, while acceptable, was inferior to that obtained with the Methocel coated kraft separator of Programs 3 and 5.

The anode with batiste fabric separator has excellent corrosion patterns which were comparable to those obtained in Programs 3 and 5. We concluded that batiste fabric separator would be a good substitute for kraft paper.

After dissection of cells stored at 130°F. at this laboratory, the following was observed.

1) The anodes of cells incorporating the fiberglass cloth showed pitting and cavitations, poor contact between cloth and anode and two seconds time delay at the end of the seven weeks.
2) The anodes with batiste fabric showed no pitting during the first and second week. During the fourth week, a fine pitting appeared with approximately the same type pitting after the seventh week, but no cavitations. The time delay after seven weeks was in the order of one second. This is one of the smallest time delays observed in these test.
3) The anodes of cells employing the gauze separator showed a similar pitting to the batiste, with a time delay in the order of 2 seconds.
4) The anodes of cells using Methocel paper showed scattered cavitations in the first week, but the pitting at the end of seven weeks was less than

for anodes used with the batiste separator. The time delay after seven weeks was 3 seconds. In general, the progress of the pitting during storage was very slow.

It is concluded that there is not much difference between the gauze, the batiste, and the Methocel paper with a slight tendency for better results with the Methocel paper.

H - SHELF PROGRAM NO. 4

The data obtained from the various screening programs supports the fact that the major cause of the severe anode open circuit corrosion and initial slow voltage recovery has been the reduction of the protective chromate film to deposit cathodic chromium metal on the magnesium during hot forming of the anode. The simplest method of avoiding the chromate film reduction is to apply the film chromate after anode forming so that it never experiences the high temperature used in anode forming. In view of this data, Shelf Program No. 4 should be undertaken employing the following specifications:

A. Anode: 0.055" magnesium anode, made out of metal supplied without the Dow 21 and treated, after forming for 3 minutes in the Dow 21.

B. Separator: One layer kraft paper coated with Methocel on both sides.

C. Cathode: 50 gms. mix, type M manganese dioxide, 88-1-3-8, wet 550 ml. of electrolyte/1000 gms dry mix, consolidated at 40 lbs/in^2.

D. Electrolyte: 250 g/l $MgBr_2$ plus 0.25 g/l Na_2CrO_4.

E. Structure: 1/32" wall cup, fiberglass tape wrapping, plastic bottom, seamless steel jacket, and two paper washers.

Initial data obtained from these cells are shown in Table XV,

Table XVI and Table XVII. In general, the performances were excellent; there were signs of adverse corrosion attack and high maximum delayed action during the LIT testing probably related to the cell formulation.

The radial expansion has been controlled with the seamless steel jacket. None of the cells in this program have been dissected to observe pitting. Screening Program No. 3 had cells similar to those in this program. The high performance capacity has been very well retained after the first month on shelf.

NOTE - DATA ON CELLS MADE PRIOR TO THE CONTRACT

As reference data that could help isolate the source of pitting and high time delay, the following information is included:

Cell made in this laboratory with the following specifications:

Structure:	1/16" wall cup, scotch tape as seal, plastic jacket, and steel closure with asphalt.
Anode:	0.060" magnesium AZ 21 x 1 supplied with Dow 21, without further treatment after forming.
Separator:	Kraft paper coated with starch gel.
Cathode:	50 gms. mix 89-3-8, African ore MnO_2, wet 370 ml. of electrolyte/1000 gms. dry mix, ~~200~~ g/l $MgBr_2$ plus 50 g/l $SrBr_2$ plus 0.2 g/l Na_2CrO_4 plus 0.25% Magnesium powder.
Consolidation:	50 lbs/in^2.

New cell had O.C.V. 1.92 volts
S.C.C. 20 amperes

After three years storage at 70°F., this cell had O.C.V. 180 volts; time delay with 4 ohms resistor, 9.5 seconds. After dissection, the anode presented no visible pitting and an excellent corrosion pattern.

CONCLUSIONS

1. The plastic closure used for the bottom of the cell has markedly improved the cell capacity retention when subjected to 130°F.
2. Of the mixed bromides tested, the best performance was obtained with the Mg/Sr mixture having a normality ratio of 2/1.
3. The use of a seamless steel jacket has virtually eliminated the expansion problem.
4. The major cause of anode perforation on shelf was cathodic chromium residue resulting from chromate ion reduction during hot forming. Cleaning the anode in Dow 21 pickle after forming has markedly reduced the pitting and cavitations on the anode.
5. The rolled anode forming technique did not attenuate the pitting.
6. The problems of the pitting and time delay, although markedly reduced have not been completely eliminated.

PROGRAM FOR NEXT INTERVAL

Work under this program will continue from October 1, 1964 to April 1, 1965 under the present contract which has been extended to September 30, 1965.

During this period, our immediate program will include the following areas:

1) Shelf life and low temperature evaluation will be continued employing the "D" size cell.
2) The investigations necessary to establish the design parameters required for the construction of a "AA" size cell utilizing the reverse electrode type of dry battery structure covered by U. S. Patent 2,903,499 will be conducted.
3) Work on the construction of a special carbon cup forming machine, molds, magnesium anode forming equipment, dies, manually operated filling machine, and crimping machine for the AA size cell will be started.
4) Further work to investigate the causes responsible for the pitting and slow voltage recovery will be conducted.

IDENTIFICATION OF KEY PERSONNEL

Mr. Rodolfo Rodriguez Balaguer
Director of Research

Mr. Balaguer received his M. S. degree in Electrical Engineering from the University of Habana in 1946. He has been engaged in battery research and development activities for the last 18 years. Since 1959, he has been the Director of Research of Caribbean Trading Corporation. Three hundred forty-three (343) hours of Mr. Balaguer's time were expended on this program.

Mr. Guillermo Perez Profet
Assistant to Director of Research

Mr. Perez received his B. S. degree in Mechanical Engineering from Rensselaer Polytechnic Institute in 1954. He has been engaged in battery research and development activities for the last 7 years when he became assistant to Mr. Balaguer. Three hundred thirty-six (336) hours of Mr. Perez's time were expended on this program.

TABLE I

Contract No. DA 36-039 AMC-03369 (E)
Experimental Design Program No. 4

Effect of 130°F. Storage Time on Performance
7.5 Ohm Cont. 70°F.

Batch No.	1	2	3	4	5	6	7	8
Mix Wetness - cc's/1000 gms.	500	500	500	500	550	550	550	550
Weight - gms	47	50	52	55	47	50	52	55
Packed - lbs/in^2	20	40	20	40	20	40	20	40
Initial C.C. volts - 0 months	1.76	1.76	1.77	1.77	1.74	1.67	1.73	1.77
1 "	1.67	1.64	1.76	1.72	1.50	1.69	1.67	1.69
2 "	0.73	1.50	1.63	1.59	1.70	1.73	1.36	1.70
Average C.C. volts - 0 months	1.45	1.46	1.44	1.44	1.43	1.44	1.43	1.40
1 "	1.34	1.32	1.39	1.40	1.26	1.38	1.37	1.35
2 "	1.17	1.41	1.34	1.38	1.40	1.44	1.27	1.44
Hours to 1.0 volt - 0 months	28	29	30	34	28	30	31	32
1 "	32	42	38	42	37	36	40	42
2	30	28	32	33	28	27	32	31
% Cathode Utilization - 0 months	79	78	78	79	79	81	82	81
1 "	86	103	96	100	97	101	100	103
2 "	67	72	73	75	75	73	73	76
Expansion - Inches x 10^3 radial - 0 months		5	19	18	7	16	14	12
1 "	21	31	25	28	29	29	20	23
2 "	12	14	26	19	23	21	20	20
Longitudinal - 0 months	1	1	0	1	4	1	1	0
1 "	5	2	2	2	2	2	0	3
2 "	26	0	1	1	0	1	1	1

TABLE II

Contract No. DA 36-039 AMC 03369 (E)
Experimental Design Program No. 4

Effect of Variables After 2 Months at 130° F.
7.5 Ohm Cont. at 70° F.

Effect of Mix Wetness	Hours to 1.0 Volt	%Cathode Utilization	Radial Exp. In. x 10^3
500	31	72	18
550	30	74	21
Effect of Mix Height			
A	28	72	18
B	32	74	21
Effect of Mix Pressure			
20 lbs/in^2	30	72	20
40 lbs/in^2	30	74	20

TABLE III

Contract No. DA 36-039 AMC-03369 (E)
Experimental Design Program No. 5

Initial Low Temperature Discharge Data for Mixed Bromide Cells

Cell Lot No.	Cell No.	Service to 0.9V through 7.5 Ohms at -20°F. in Min.	Delayed Action in Min. to 0.9 V.	Cell No.	Service to 0.9 V through 2.25 Ohms at -20°F.	Cell No.	Service to 0.9V through 7.5 Ohms at +20°F. in hours	Delayed Action in Min. to 0.9 V	Cell No.	Service to 0.9V through 2.25 Ohms at +20°F. in Min.	Delayed Action in Min. to 0.9V
1	446	78	5	443	0	445	18.9	>0.5	444	312	>0.1
2	448	71	6	450	0	449	20.3	>0.5	447	260	0.7
3	453	84	8.5	452	0	454	19.1	>0.5	451	250	2.0
4	458	214	1.2	455	0	456	20.9	>0.1	457	330	>1.0
5	462	142	2.9	460	0	461	22.3	>0.5	459	290	>1.0
6	464	151	8.0	463	0	466	24.4	>0.1	465	280	>1.0
7	470	0	--	467	0	468	21.0	>0.1	469	201	>1.0
8	472	1	>1	474	0	473	22.0	>0.1	471	60	>1.0
9	477	6	>1	476	0	475	17.2	>0.5	478	2	0.5
10	481	0	--	480	0	482	18.5	>0.5	479	160	10.0
11	484	0	--	485	0	486	19.5	1.1	483	250	0.9
12	487	95	8	488	0	489	17.0	0.7	490	215	4.5
13	493	165	20	492	0	494	19.3	0.6	491	247	>0.5
14	496	0	--	495	0	497	21.2	0.8	498	240	>0.5
15	502	160	27	500	0	499	18.5	0.5	501	233	1.2
16	503	26	7	504	0	505	18.2	>0.3	506	170	7.5
17	510	85	5	507	0	508	19.2	>0.5	509	134	0.7
18	511	0	--	512	0	514	15.8	>0.5	513	173	>0.5

TABLE IV

Contract No. DA 36-039 AMC-03369(E)
Experimental Design Program No. 6

Initial Test at 70°F.

Batch No.	ELECTROLYTE $Mg(ClO_4)$ N	$Mg(ClO_4)$ Gm/l	Na_2CrO_4 Gms/l	2.25 OHM C.C. Volt Init.	2.25 OHM C.C. Volt Av.	2.25 OHM Min. to .90V.	2.25 OHM Exp. in. x10^3 R	2.25 OHM Exp. in. x10^3 L	7.50 OHM C.C. Volt Init.	7.50 OHM C.C. Volt Av.	7.50 OHM Hours to 1 volt	7.50 OHM % Mix Util.	7.50 OHM Exp. in. x10^3 R	7.50 OHM Exp. in. x10^3 L
1	2.0	223	0	0.94(1)	1.29	405	1	0	1.17(1)	1.32	16(3)		0	0
2	3.5	391	0	1.13(1)	1.32	460	8	0	1.28(1)	1.43	25		0	0
3	5.0	558	0	1.64	1.22	425(2)	17	0	1.58(1)	1.48	25(2)		5	0
4	2.0		.25	1.03(1)	1.37	400	4	0	1.56(1)	1.44	25		2	
5	3.5		.25	1.23(1)	1.33	450	5	0	1.69(1)	1.48	25		1	
6	5.0		.25	1.58	1.34	395(2)	16	0	1.47(1)	1.46	25(2)		9	

CATHODE: Type M Ore 88-1-3-8 Wet 620 cc's Elect./1000 gms.
Dry 55 gms/Cell Consolidated at 40 lbs/in^2
ANODE: AZ 21 x 1 .055"
SEPARATOR: Kraft paper - 5% Methocel coated - 2 sides
STRUCTURE: Fiberglass Tape Wrapped - Steel Jacket with Seam-Koldmount Closure

(1) Initial Voltage lower than 15 min. reading
(2) Seam Split
(3) Loss of contact

TABLE V

Contract No. DA 36-039 AMC-03369 (E)
Experimental Design Program No. 6

Initial Low Temperature Discharge Data

Cell Lot No.	Cell No.	Service to 0.9 V through 7.5 ohms at -20°F. in hours	Delayed Action in Min. to 0.9 V	Cell No.	Service to 0.9V through 2.25 ohms at -20°F. in hours	Cell No.	Service to 0.9V through 7.5 ohms at +20°F. in hours	Delayed Action in Sec. to 0.9 V	Cell No.	Service to 0.9V through 2.25 ohms at +20°F. in hours	Delayed Action in Sec. to 0.9 V
1	600	3.1	12.1	602	0	603	21.0	170	601	3.6	330
2	612	6.3	13.1	609	0	610	24.5	28	611	2.9	110
3	621	9.0	135.0	619	0	618	14.0	14	620	2.2	40
4	629	3.4	101.0	630	0	627	15.0	65	628	4.0	67
5	638	5.9	425	637	0	636	21.0	36	639	3.0	16
6	646	8.3	2.4	647	0	648	13.0	20	645	1.4	45

TABLE VI

Contract No. DA36-039 AMC 03369(E)

Caribbean Trading Corporation Test Results
High Temperature Test 130°F.
7.5 Ohms Cont. Test at 70°F.

Cell No.	Storage	C. C. V.	Hours to 1.0V.	Watt-hr/lb.	Ret. Cap.	Leakage
MG2-649	Fresh	1.75	29.50	32.7	--	No
MG2-650	"	1.72	31.00	35.5	--	No
Avg.		1.73	30.25	34.1		
MG2-651	1 month	1.66	31.30	34.4	104.16%	No
MG2-652	"	1.60	30.30	31.0	100.83%	No
Avg.		1.63	31.00	32.7	102.49%	
MG2-661	2 month	1.46	29.50	33.5	97.52%	No
MG2-662	"	1.52	28.50	29.5	94.22%	No
Avg.		1.49	29.00	31.5	95.87%	
MG2-655	3 month	1.62	30.25	31.8	100.00%	No
MG2-656	"	1.67	29.30	31.4	97.52%	No
Avg.		1.64	29.77	31.6	98.76%	
MG2-657	4 month	1.60	32.00	36.8	105.78%	No
MG2-658	"	1.10	26.25	26.8	86.78%	No
Avg.		1.35	29.12	31.8	96.28%	
MG2-659	5 month	1.62	26.50	26.2	88.50%	No
MG2-660	"	1.52	18.00	16.6	59.50%	No
AVG		1.57	21.25	21.4	74.00%	

TABLE VII

Contract No. DA36-039 AMC 03369(E)

Caribbean Trading Corporation Test Results
High Temperature Test 165°F.
7.5 Ohms Continuous Test at 70° F.

Cell No.	Storage	C. C. V.	Hours to 1.0 V.	Watt-hrs/lb.	Ret. Cap.	Leakage
MG2-649	Fresh	1.75	29.50	32.7	--	No
MG2-650	Fresh	1.72	31.00	35.5	--	No
Avg.		1.73	30.25	34.1		
MG2-667	1 month	1.70	29.00	29.3	96.88%	No
MG2-668	1 month	1.48	27.30	27.8	91.95%	No
Avg.		1.59	28.15	28.5	94.41%	
MG2-669	2 month	1.20	21.00	17.6	67.00%	No
MG2-670	2 month	1.42	25.00	23.4	82.65%	No
Avg.		1.31	23.00	20.5	74.82%	
MG2-671	3 month	.87	16.00	14.04	46.30%	
MG2-672	3 month	1.32	24.25	17.37	80.17%	
Avg.		1.09	20.12	15.70	63.23%	

TABLE VIII

Contract No. DA 36-039 AMC-03369 (E)

Caribbean Trading Corporation Test Results
High Temperature Test 165°F.
7.5 Ohms Continuous Test at 70 °F.

Cell No.	Storage	C.C.V.	Hours to 1.0V.	Watt-hr/lb.	Ret. Cap.	Leakage
MG2-515	Fresh	1.84	31.75	38.4	--	No
MG2-516	Fresh	1.84	31.75	38.4	--	No
Avg.		1.84	31.75	38.4		
MG2-517	15 days	1.83	29.5	31.2	92.9%	Yes
MG2.518	15 days	1.79	31.5	32.8	99.2%	No
Avg.		1.81	30.5	32.0	96.0%	
MG2-519	1 month	1.76	31.25	34.8	98.4%	Yes
MG2.520	1 month	1.76	28.25	32.7	89.1%	Yes
Avg.		1.76	29.75	33.7	93.7%	
MG2-521	2 months	1.70	22.25	23.4	70.0%	No
MG2-522	2 months	1.70	23.00	25.7	72.4%	No
Avg.		1.70	22.62	24.5	71.2%	
MG2-523	3 months		CELLS DEAD	DRIED OUT		
MG2-524	"					

1/32" Wall Cup
55 Grs. Mix, type M MnO_2, wet 550 gms. of electrolyte/1000 gms dry mix
40 lbs/in^2 consolidation
0.055" Mag. AZ 21 x 1
Fiberglass tape, plastic bottom, steel jacket with seam
Electrolyte: 250 g/l $MgBr_2$ plus 0.25 g/l Na_2CrO_4

TABLE IX

Contract No. DA 36-039 AMC 03369(E)

Caribbean Trading Corporation Test Results
High Temperature Test 130°F.
7.5 Ohms Continuous Test at 130°F.

Cell No.	Storage	C.C.V.	Hours to 1.0V	Watt-hr/lb.	Ret. Cap.	Leakage
MG2-515	Fresh	1.84	31.75	--	--	No
MG2-516	"	1.84	31.75	--	--	No
		1.84	31.75	38.4		
MG2-525	1 month	1.80	32.0	37.4	100.8%	No
MG2-526	1 month	1.79	32.5	38.6	102.4%	No
Avg.		1.79	32.3	38.0	101.6%	
MG2-527	2 months	1.72	30.5	37.3	96.0%	No
MG2.528	2 months	1.74	28.0	33.9	88.2%	No
Avg.		1.73	29.2	35.6	92.1%	
MG2-530	3 months	1.76	31.5	39.7	99.20%	No
MG2-531	3 months	1.81	30.5	34.7	96.06%	No
Avg.		1.78	31.0	37.2	97.54%	

1/32" Wall Cup
55 grs-mix, Type M MnO_2, wet 550 gms./1000 gms dry mix
40 lbs/in^2 Consolidation
0.055" Magnesium AZ 21 x 1
Fiberglass tape, plastic bottom, steel jacket with seam
Electrolyte: 250 g/l $MgBr_2$ plus 0.25 g/l Na_2CrO_4

TABLE X

Contract No. DA36-039 AMC 03369(E)

Shelf Life Program No. 3
Initial Capacity Data

2.25 Ohm Cont.						7.5 Ohm Cont.						
	C.C. Volt		Minutes to	Expansion in. x 10^3			C.C. Volt		Hours to	% Mix	Expansion in. x 10^3	
Cell No.	Init.	Av.	0.9 volt	Radial	Length	Cell No.	Init.	Av.	1.0volt	Utilis.	Radial	Length
539(1)	1.52(3)	1.35	480	3	1	532(1)			Dead			
561	1.39(3)	1.32	450	3	0	543(2)	1.78	1.48	31		18	
570	1.55(3)	1.38	480	4	0	563(2)	1.82	1.49	31		12	
540	1.55(3)	1.41	390	2	0	567(2)	1.62(3)	1.45	28		9	
534	1.21(3)	1.30	450	0	0	574(2)	1.55(3)	1.43	29		15	
AV.		1.35	450	2	0			1.46	30	72	14	

BA 30 Test
6-6 Ohms

Cell No.	C.C. Volt Init.	Av.	Days to .93 Volt	Delay to .93 volt Seconds
535	1.70	1.40	31	3.6 Max.
547	1.77	1.46	32	4.8 Max.
576	1.70	1.48	31	4.8 Max.

Batch 8 - Experimental Design Prog. No. 4
Steel Jacket over Fiberglass tape wrap - kraft paper, Methocelcoated - Type M Ore 88-1-3-8, wet 550 with 250 g/l $MgBr_2$ - 55 gms. of mix.

(1) Seam Bulged
(2) Seam Split
(3) Initial Voltage (1 minute) Lower than 15 minute reading.

TABLE XI

Contract No. DA36-039 AMC-03369(E)

Screening Program No. 1
Separator Study

Cell No.	SEPARATOR No. of layers	SEPARATOR Method Applied	Flash Current Amps	C. C. Volts at 7.5 ohms	Delay Seconds to C. C. volts	Anode Corrosion Pattern
682	1	New	1.8	1.60	15	Large percentage of surface perforated
683	1	New	2.7	1.75	10	
694	2	New	4.6	1.75 (a)	5	Moderate to heavy Pitting
695	2	New	4.5	1.78	7.5	
706	3	New	4.4	1.78	--	Heavy Pitting
707	3	New	5.4	1.76 (b)	8	
718	1	Old	6.0	1.80 (a)	9.5	
719	1	Old	6.7	1.80	7.3	Moderate Pitting

(a) Heavy Pitting Along Butt seam
(b) Heavy Pitting at Top of Anode

TABLE XII

Contract No. DA 36-039 AMC 03369(E)

Screening Program No. 2
Consolidateion Study
7.5 Ohms Continuous Test at 70°F.

INITIAL 7.5 OHM CONT.						1 WEEK AT 113°F.				
			Delay Data					Delay Data		
Cell No.	* Separator	Initial C.C. volt.	Min. volt.	Sec. to 1.0 V.	Hours Cap.	Cell No.	Initial C.C. volt	Min. Volt	Sec. to 1.0 V.	Hours Cap.
730	1	1.65	0.45	1.0	28.6	732	1.65	0.17	2.8	28.0
731	1	1.60	0.25	1.9	28.1	733	1.60	0.25	4.3	29.1
742	2	1.64	0.35	1.8	27.0	744	1.73	0.25	2.9	26.8
743	2	1.75	0.40	2.8	25.7	745	1.76	0.30	5.3	25.9
754	3	1.73	0.40	2.5	26.5	756	1.75	0.25	2.8	21.2
755	3	1.71	0.35	3.0	27.5	757	1.73	0.25	3.3	17.8
766	4	1.65	0.25	1.4	29.6	768	1.75	0.23	3.5	29.5
767	4	1.70	0.25	0.8	27.8	769	1.45	0.30	8.4	29.2

* 1 Layer applied one piece
2 Layers applied one piece
3 Layers applied one piece
4 1 layer applied 4 overlapping strips

TABLE XIII

Contract No. DA36-039 AMC-03369(E)
Experimental Design Program No. 7
Initial Capacity Data
"D" Size Cells Discharged thru 7.5 ohms

Cell No.	Electrolyte	CC/1000 Wet	G/Cell	Delay		Flash Current Amps	Discharge		
				Min. Voltage	Sec. to 1.0 V.		Init. Volts	Av. Volts	Hrs. to 1.0 V.
235327-778	$MgBr_2$	490	40	0.35	2.6	3.8	1.75	1.45	17
328-784	"	490	50	0.45	3.3	4.5	1.75	1.50	21.8
329-790	$Mg(ClO_4)_2$	490	40	0.38	5.8	2.8	--	1.34	13.3
330-796	"	490	50	0.37	3.2	4.6	1.82	1.42	20.7*
331-802	$Mg(ClO_4)_2$	550	40	0.24	3.7	3.2	1.78	1.40	14.3*
332-808	"	550	50	0.15	3.5	3.3	1.78	1.46	25.5*

*Bottom closure pushed out & cell leaked during discharge

TABLE XIV

Contract No. DA 36-039 AMC-03369 (E)

Screening Program No. 7
Separator Studies
7.5 Ohms Test at 70°F.

	INITIAL 7.5 OHM DATA					1 week at 113° F.			
			Delay Data				Delay Data		
	Cell No.	C. C. V.	Min. volts	Sec. to 1.0 V.	Flash Current Amperes	Cell No.	Min. volts	Sec. to 1.0 V.	Flash Current Amperes
Methocel Paper	897	0							
	898	1.70	.50	0.8	4.8		.25	2.4	4.1
Fiberglass	909	1.10	.45	200	1.8	910	.35	6.6	0.6
2 layers Sterile Gauze	920	1.75	.40	0.5	7.1	921	.30	1.5	4.3
Batiste Fabric	932	1.75	.43	0.3	6.5	933	.30	0.4	6.0

TABLE XV

Contract No. DA 36-039 AMC 03369 (E)
Shelf Program No. 4

Initial Capacity Data at 70°F.

2.25 Ohms Cont.

Cell No.	O.C.V.	C.C.V.	Delay Data: Min. Volt.	Delay Data: Sec. to 1.0 V.	Av. Volt.	Hours to 0.90 V.	Flash Current
1	1.86	1.72	.20	0.5	1.37	7.0	8.1
2	1.86	1.70	.15	0.6	1.36	7.5	7.8
3	1.88	1.72	.15	0.8	1.41	6.3	8.8
4	1.84	1.70	.30	3.3	1.40	6.6	7.2
5	1.86	1.72	.25	0.9	1.41	7.3	8.1
Av.	1.86	1.71	.21	1.2	1.39	6.94	8.0

7.5 Ohms Cont.

						Hours to 1.0 V.	
6	1.86	1.78	.45	1.2	1.47	31.0	7.8
7	1.88	1.79	.40	0.5	1.51	28.3	8.8
8	1.87	1.81	.35	0.5	1.49	29.5	8.6
9	1.88	1.81	.40	0.5	1.50	29.2	9.4
10	1.88	1.81	.35	0.3	1.52	28.4	8.8
Av.	1.87	1.80	.39	0.6	1.50	29.3	8.7

TABLE XVI

Contract No. DA 36-039 AMC-03369 (E)

Shelf Program No. 4
Initial Capacity Data at 70 °F.
2.25 Ohm Light Industrial Test.

Cell No.	Voltage O.C.	Voltage C.C.	Delay Data - Sec. to .90 V. AM AV.	AM Max.	PM AV.	PM Max.	Av. Volt.	Minutes Cap. to .90V.	Amps Flash Current
11	1.88	1.72	1.0	5.8	0.7	3.7	1.42	598	8.7
12	1.88	1.71	1.1	5.7	0.6	3.1	1.42	573	8.6
13	1.88	1.70	0.8	6.3	0.6	3.6	1.38	603	7.8
14	1.86	1.66	0.8	6.0	0.6	3.2	1.38	620	8.0
15	1.87	1.64	2.0	11.0	0.6	3.2	1.38	559	7.0
Av.	1.87	1.69	1.1		0.6		1.40	590	8.0

AM Delay taken prior to first discharge of the day
PM " " " " last " " " " "
Delays taken with 2.25 Fixed Res.

TABLE XVII

Contract No. DA 36-039 AMC 03369 (E)

Caribbean Trading Corporation Test Results
Shelf Life Program No. 4
130°F. Storage Temperature
7.5 Ohm Continuous Test at 70° F.

Cell No.	Storage	Delay Data		C.C.V.	Hours to 1 V.	Watt-hr/lb.	Ret. Cap.	Leakage	Exp. in. x 10^3	
		Min. V	Sec. 1V						Rad.	Leng.
MG2-1020	Fresh	No	Lessthan 1	1.86	29.5	38.3	--	No	0	0
MG2-1021	Fresh	No	sec.	1.84	29.5	38.3	--	No	0	0
Av.		No		1.85	29.5	38.3			0	0
MG2-1022	1 month	No	Less than	1.81	29.75	35.40	100.85%	No	0	0
MG2-1023	"	No	1 sec.	1.83	28.30	33.60	96.60%	No	0	0
		No		1.82	29.02	34.50	98.72%		0	0

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LOW TEMPERATURE BATTERY
(NEW MAGNESIUM ANODE) **R. R. Balaguer**
Semi-Annual Report No. 4 **15 tables**
October 31, 1964 31 pages
1 April 1964 to 30 September 1964

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DA Proj. No. 1C6-22001-A.053
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1. Primary Cells
2. Magnesium dry cells (low temperature)
3. Balaguer reversed electrode structure

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